CLAIMS

1. A process for preparation and isolation of a non-amorphous cationic rhodium complex of formula (1), wherein ligand represents an enantiomerically enriched organic compound possessing one or two ligating phosphorus atoms, and wherein m = 2 when the ligand is monodentate and m = 1 when the ligand is bidentate, which comprises the following steps:

(a) Dissolution of Rh(diolefin)(acac) in one or more ethereal solvents;
(b) Addition of a fluorinated non-mineral acid HX and alcohol solvent or alcohol-containing solvent mixture, either simultaneously or sequentially, to form a soluble solvated complex of rhodium with one or more of the reaction solvents;
(c) Addition of the ligand, either in solution in an organic solvent or neat;
(d) Collection of the crystalline precipitate of complex (1).

$$[Rh(ligand)_m(diolefin)]^+ X^- \quad (1)$$

2. A process according to claim 1, wherein step (b) comprises simultaneous addition of HX and alcohol solvent or alcohol-containing solvent mixture.

3. A process according to claim 2, wherein step (b) comprises addition of HX as a solution in an alcohol solvent or alcohol-containing solvent mixture.

4. A process according to claim 1, wherein step (b) comprises sequential addition, in either order, of HX and alcohol solvent or alcohol-containing solvent mixture.

5. A process according to claim 1, wherein the diolefin is a cyclic diolefin.

6. A process according to claim 5, wherein the diolefin is either 1,5-cyclooctadiene (COD) or 2,5-norbornadine (NBD).

7. A process according to claim 6, wherein the diolefin is COD.

8. A process according to claim 1, wherein diolefin represents two molecules of an olefin selected from the group consisting of ethylene and C_{5-10} cycloalkenes.

9. A process according to claim 1, wherein HX is a perflourinated non-mineral acid.

10. A process according to claim 9, wherein HX is selected from the group consisting of HBF_4, HPF_6, $HSbF_6$ and CF_3SO_3H.

11. A process according to claim 10, wherein HX is HBF_4.

12. A process according to claim 1, wherein ethereal solvents are selected from the group consisting of dialkyl ethers, tetrahydrofuran, 1,4-dioxane and 1,2-dimethoxyethane.

13. A process according to claim 12, wherein dialkyl ethers are selected from the group consisting of *t*-butyl methyl ether, diethyl ether, diisopropyl ether and di-*n*-butyl ether.

14. A process according to claim 13, wherein a dialkyl ether is in admixture with tetrahydrofuran.

15. A process according to claim 14, wherein the ratio of dialkyl ether:tetrahydrofuran ranges from about 10:1 to about 1:1.

16. A process according to claim 15, wherein the ratio of dialkyl ether:tetrahydrofuran ranges from about 6:1 to about 2:1.

17. A process according to claim 16, wherein the dialkyl ether is *t*-butyl methyl ether.

18. A process according to claim 1, wherein the alcohol is a linear or branched C_{1-6} alkanol.

19. A process according to claim 18, wherein the alkanol is selected from the group comprising methanol, ethanol, n-propanol, isopropanol, and 1-butanol.

20. A process according to claim 1, wherein the organic solution used for dissolution of ligand is selected from the group comprising ethereal solvents, non-polar hydrocarbon solvents and mixtures thereof.

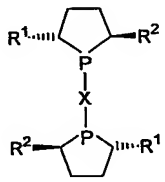
21. A process according to claim 1, wherein m = 1.

22. A process according to claim 21, wherein the ligand is a diphosphine.

23. A process according to claim 22, wherein the diphosphine is a bisphosphacycle.

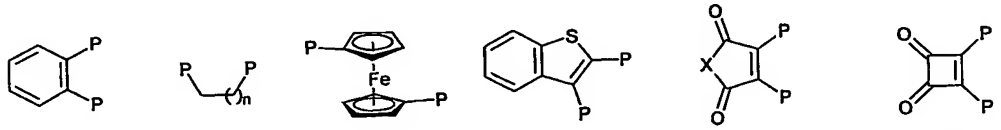
24. A process according to claim 23, wherein the bisphosphacycle is a bisphospholane.

25. A process according to claim 24, wherein the bisphosphacycle is a bisphospholane according to formula (**2**), or the opposite enantiomer thereof, wherein X represents an organic or organometallic bridging radical, R^1 and R^2 are each independently H or an optionally substituted hydrocarbon group, provided that R^1 and R^2 are not both H, and the 3- and 4-positions of either or both phospholane rings optionally may be substituted with one or more non-interfering groups.



(**2**)

26. A process according to claim 25 wherein P-X-P in the bisphospholane is selected from a group consisting of formulae (**3**) to (**8**), each of which may be optionally substituted; n in (**4**) is in the range 0-5; X in (**8**) is either O or N-alkyl.



(**3**) (**4**) (**5**) (**6**) (**7**) (**8**)

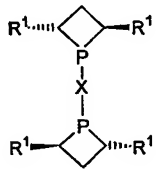
27. A process according to claim 26, wherein P-X-P is of formula (**3**).

28. A process according to claim 26, wherein P-X-P is of formula (**4**) and n = 1.

29. A process according to claim 26, wherein P-X-P is of formula (**5**).

30. A process according to claim 24, wherein the bisphosphacycle is a bisphospholane according to formula (**9**), the opposite enantiomer thereof and substituted analogues thereof.

31. A process according to claim 23, wherein the bisphosphacycle is a bisphosphetane of formula (**10**), wherein X represents an organic or organometallic bridging radical, R^1 and R^2 are each independently H or an optionally substituted hydrocarbon group, provided that R^1 and R^2 are are not both H, and the 3-position of either or both phosphetane rings optionally may be substituted with one or more non-interfering groups.



(**10**)

32. A process according to claim 31, wherein X is 1,1'-ferrocenyl.

33. A process according any of claims 25-29 or 31-32, wherein R^1 and R^2 are each independently $C_{1\text{-}20}$ alkyl, aryl or aralkyl.

34. A process according to claim 33, wherein $R^1 = R^2 = C_{1\text{-}20}$ alkyl.

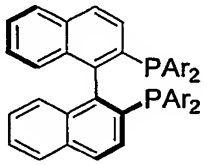
35. A process according to claim 34, wherein alkyl is selected from the group consisting of methyl, ethyl, *n*-propyl, isopropyl, *n*-butyl, isobutyl, *sec*-butyl and *t*-butyl.

36. A process according to claim 33, wherein $R^1 = R^2$ = phenyl.

37. A process according to claim 19, wherein the diphosphine is an atropisomeric diphosphine containing two $P(Ar)_2$ groups, wherein Ar = phenyl, optionally substituted with one or more alkyl or alkoxy groups.

38. A process according to claim 32, wherein the diphosphine is a biaryldiphosphine.

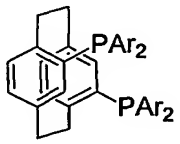
39. A process according to claim 33, wherein the biaryldiphosphine is a BINAP ligand of formula (**11**), or the opposite enantiomer thereof.



(**11**)

40. A process according to claim 38, wherein the biaryl moiety is heteroaromatic.

41. A process according to claim 32, wherein the diphosphine is a PHANEPHOS ligand of formula (**13**), or the opposite enantiomer thereof.



(**13**)

42. A process according to claim 21, wherein at least one of the ligating phosphorus atoms in the ligand is covalently bonded to one or more heteroatom.

43. A process according to claim 42, wherein both ligating phosphorus atoms are covalently bonded to one or more heteroatoms.

44. A process according to claim 43, wherein the ligand is selected from the group consisting of bisphosphites, bisphosphinites, bisphosphonites and bisphosphoramidites.

45. A process according to claim 1, wherein m = 2.

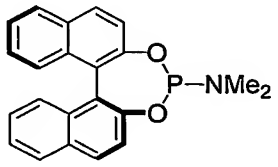
46. A process according to claim 45, wherein the ligand is a monophosphine.

47. A process according to claim 46, wherein the phosphine is a *P*-aryl phosphacycle.

48. A process according to claim 45, wherein the ligating phosphorus atom in the ligand is covalently bonded to one or more heteroatoms.

49. A process according to claim 48, wherein the ligand is a phosphoramidite.

50. A process according to claim 49, wherein the phosphoramidite is of formula (**14**) or the opposite enantiomer thereof.



(**14**)

51. A process according to claim 1, wherein the complex (**1**) is prepared directly from a ligand precursor containing one or more acid-labile hydroxyl protecting groups, which are removed during complex formation.

52. A process according to claim 1, wherein the complex (**1**) is obtained is in a crystalline form.

53. A process according claim 1, wherein the complex (**1**) is stable to storage, under an inert atmosphere at ambient temperature, for at least three (3) days.

54. A process according the claim 1, wherein the ligand is enantiomerically enriched to at least 95% ee.

55. A process according the claim 54, wherein the ligand is enantiomerically enriched to at least 99% ee.

56. A process according to claim 55, wherein the ligand is enantiomerically pure.